

AFRL-PR-WP-TP-2006-215

**COPPER METALLIC SUBSTRATES
FOR HIGH TEMPERATURE
SUPERCONDUCTING COATED
CONDUCTORS**



**Nicholas A. Yust, Rama Nekkanti, Lyle B. Brunke, Raghavan Srinivasan,
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APRIL 2006

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. REPORT DATE (DD-MM-YY) April 2006			2. REPORT TYPE Journal article postprint		3. DATES COVERED (From - To) 04/14/2003 – 04/14/2004	
4. TITLE AND SUBTITLE COPPER METALLIC SUBSTRATES FOR HIGH TEMPERATURE SUPERCONDUCTING COATED CONDUCTORS			5a. CONTRACT NUMBER In-House			
			5b. GRANT NUMBER			
			5c. PROGRAM ELEMENT NUMBER 61102F/62203F			
6. AUTHOR(S) Nicholas A. Yust, Lyle B. Brunke, and Paul N. Barnes (Power Generation Branch (PRPG), Power Division) Rama Nekkanti (UES, Inc.) Raghavan Srinivasan (Wright State University)			5d. PROJECT NUMBER 3145			
			5e. TASK NUMBER 32			
			5f. WORK UNIT NUMBER 314532Z9			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Power Generation Branch (PRPG), Power Division Propulsion Directorate Air Force Materiel Command, Air Force Research Laboratory Wright-Patterson AFB, OH 45433-7251			8. PERFORMING ORGANIZATION REPORT NUMBER AFRL-PR-WP-TP-2006-215			
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Propulsion Directorate Air Force Research Laboratory Air Force Materiel Command Wright-Patterson AFB, OH 45433-7251			10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL-PR-WP			
			11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-PR-WP-TP-2006-215			
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.						
13. SUPPLEMENTARY NOTES Published in IOP Publishers' <i>Superconductor Science and Technology</i> , 18 (2005) 9-13. ©2005 IOP Publishing Ltd.						
14. ABSTRACT Biaxially cube textured polycrystalline Cu(200) substrate tapes were produced for high temperature superconducting (HTS) coated conductor applications. A comparison is made between Cu substrates fabricated by reverse cold rolling followed by recrystallization, from stock materials that were obtained in the form of extruded rod and rolled plate. Detailed x-ray diffraction (XRD) studies and orientation imaging microscopy (OIM) were performed to measure the in-plane alignment, out-of-plane alignment, and microtexture at various deformation levels and annealing temperatures. The rod starting geometry proved to have superior biaxial alignment with a predominant (220) deformation texture after rolling. Phi (Φ) scan and psi (Ψ) scan XRD reveals that the best in-plane and out-of-plane alignment, measured in terms of full width half maximum (FWHM) values of 5.4 deg. and 5.8 deg., were obtained at 99.5% reduction in thickness and 750 deg. C annealing temperature. OIM microtexture results indicate that more than 97.5% of grains had less than 10 deg. misorientation with no observable twinning.						
15. SUBJECT TERMS copper substrate, HTS, superconductor						
16. SECURITY CLASSIFICATION OF: a. REPORT Unclassified		b. ABSTRACT Unclassified		c. THIS PAGE Unclassified		
17. LIMITATION OF ABSTRACT: SAR		18. NUMBER OF PAGES 12		19a. NAME OF RESPONSIBLE PERSON (Monitor) Dr. Paul N. Barnes		
				19b. TELEPHONE NUMBER (Include Area Code) (937) 255-6241		

Copper metallic substrates for high temperature superconducting coated conductors

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Received 14 April 2004, in final form 2 August 2004

Published 16 November 2004

Online at stacks.iop.org/SUST/18/9

Abstract

Biaxially cube textured polycrystalline Cu(200) substrate tapes were produced for high temperature superconducting (HTS) coated conductor applications. A comparison is made between Cu substrates fabricated by reverse cold rolling followed by recrystallization, from stock materials that were obtained in the form of extruded rod and rolled plate. Detailed x-ray diffraction (XRD) studies and orientation imaging microscopy (OIM) were performed to measure the in-plane alignment, out-of-plane alignment, and microtexture at various deformation levels and annealing temperatures. The rod starting geometry proved to have superior biaxial alignment with a predominant (220) deformation texture after rolling. Phi (Φ) scan and psi (Ψ) scan XRD reveals that the best in-plane and out-of-plane alignment, measured in terms of full width half maximum (FWHM) values of 5.4° and 5.8°, were obtained at 99.5% reduction in thickness and 750 °C annealing temperature. OIM microtexture results indicate that more than 97.5% of grains had less than 10° misorientation with no observable twinning.

1. Introduction

Progress in metallic tape development for high J_c superconducting films has been significant over the past several years [1, 2]. Thermomechanical processing by rolling and subsequent annealing, such as the rolling assisted biaxial textured substrate (RABiTS™) process, has been successful for inducing both in-plane and out-of-plane alignment in textured metallic substrates. Previous work on textured substrates for high temperature superconducting (HTS) coated conductors has largely focused on the development of biaxially textured metallic substrates of Ni with subsequent epitaxial depositions of oxide buffer layers [3–5]. More recent studies have demonstrated the successful use of cube textured Ni alloys, such as Ni–W, as epitaxial templates for high critical current density (J_c) coated conductors [6]. Other investigations of Ni-based alloys including Ni–Cu, Ni–Cr, and Ni–V have proven successful for the (200) cube

texture, but have inconsistent J_c values for the YBCO coated conductors [7–9]. In general the Ni alloys have proven most successful for texture and compatibility with subsequent deposition techniques and commonly used oxide buffers.

For power system applications, non-magnetic metallic substrates, which minimize the AC losses experienced in many power applications, are preferred. If the substrate has high thermal and electrical conductivity, it can offer electrical stability if a conductive buffer can be successfully applied. Textured metallic substrates based on copper meet these requirements since they are non-magnetic and highly conductive. Copper also offers a low cost alternative to Ni and Ni-based alloys, since Cu is significantly less expensive, and a sharp cube texture can be obtained [10, 11].

An adverse effect of using Cu as a substrate material relates to its chemical stability, since copper oxide forms readily under ambient conditions, especially during the processing of subsequent oxide layers. Other disadvantages include

low mechanical strength, which is a concern for long wire processing. Alloying or dispersion strengthening of the copper can improve its mechanical strength but will also degrade its high conductivity. As such, proper care must be taken to achieve a proper trade-off. Overcoming the adverse affects of Cu is possible by processes such as Ni plating by epitaxial electrodeposition or sputtering [12]. Plating of Ni or other metals can prevent oxygen diffusion and increase mechanical strength. The present work involves only the fabrication of highly textured metallic substrates based on copper by reverse cold rolling followed by annealing.

2. Materials and experimental procedure

In this study, two different starting materials were used to create substrates: ASTM grade 101000 extruded copper rod and ASTM grade 10100 rolled copper plate. Both were obtained from Goodfellow and are unannealed with the copper rod (item CU007955) being as-drawn and the copper plate (item CU000766) being as-rolled. The extruded copper rod, with a diameter of $9.5\text{ mm} \pm 10\%$, was cut into samples with lengths of $100\text{ mm} \pm 5\%/-1\%$. The samples were partially flattened from 9.5 to 7.5 mm on a uniaxial forge press. This created two faceted surfaces in the axial direction which was necessary due to the restricted opening of the rolling mill. The rolled copper plate, which had a starting thickness of $2.0\text{ mm} \pm 10\%$, was cut into samples of length $100\text{ mm} \pm 2\%/-1\%$ along the rolling direction and width 20 mm.

Prior to rolling, the faceted surfaces of the copper rod and copper plate were ground successively with 120, 240, 400, and 600 grit SiC paper, and then polished to a $6\text{ }\mu\text{m}$ finish using diamond paste on microcloth. Cold work induced during these preparation steps was removed by annealing at $450\text{ }^\circ\text{C}$ for one hour in a 300 mTorr Ar/5% H atmosphere.

The copper rod and copper plate were reverse cold rolled at room temperature on a two-high mill using polished 3 inch (75 mm) diameter rolls. Both the rod and plate were rolled in steps of 10% reduction per pass, producing five final deformation levels of 95.5%, 97.5%, 98.5%, 99.0%, and 99.5%. Between 27 and 50 passes were required to reach these deformation levels, resulting in substrates that ranged in thickness from 300 to $36\text{ }\mu\text{m}$. At each deformation level, samples were taken to gauge the presence of deformation texture, and then heat treated. The samples were cut to 10 mm by 5 mm, with the long direction aligned with the rolling direction. All samples were ultrasonically cleaned with acetone and ethanol for lubricant removal.

Heat treatments were performed in a Lindberg 3 inch quartz tube furnace in a reducing atmosphere of Ar with 5% H₂. The temperature stability of the furnace is $\pm 2\text{ }^\circ\text{C}$. The furnace was heated rapidly to the annealing temperature so that each sample could reach the heat treatment temperature before significant recrystallization of the sample occurred. Samples from each of the five strain levels for the two starting configurations were annealed at temperatures in the range of 300–800 °C. An initial annealing for 6 h at a temperature of 300 °C was used for both materials. For all other temperatures the annealing time was 1 h. The samples were allowed to cool to room temperature in the furnace. The deformation levels

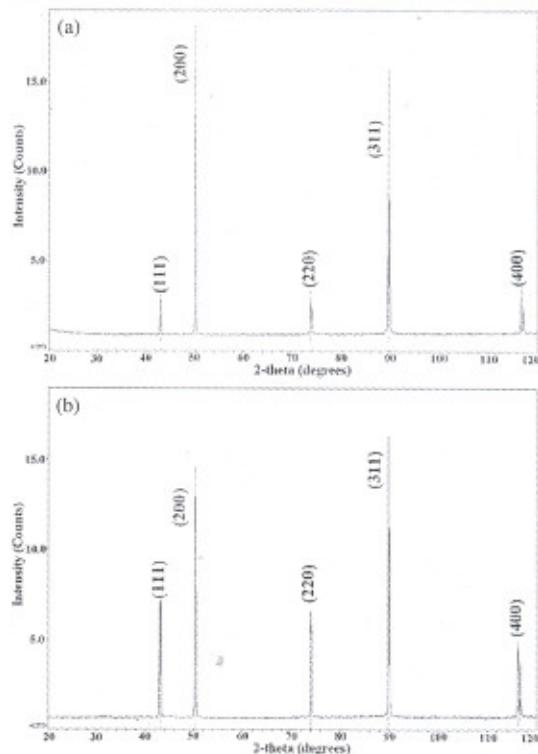


Figure 1. Texture in annealed starting materials prior to rolling. (a) 2θ scan of the copper rod, showing all dominant FCC peaks, and (b) 2θ scan of the copper plate, showing all dominant FCC peaks.

and annealing schedule for the copper rod and copper plate are summarized in table 1.

The recrystallization texture presence was analysed using 2θ scans, the in-plane and out-of-plane alignment was measured using Φ and Ψ scans, and the microtexture was characterized utilizing orientation imaging microscopy (OIM). Texture development in all samples was measured by the x-ray reflection technique utilizing Cu K α_1 radiation on a Rigaku coupled 2θ goniometer to obtain 2θ scans, and on a Phillips Materials Research Diffractometer to obtain Φ and Ψ scans. The 2θ scans were conducted between 10° and 120° with a step size of 0.05° and a scan rate of 6° min^{-1} .

3. Results and discussion

Figure 1 shows the 2θ scans for the starting copper rod and copper plate materials. Since there is no dominant peak for the plate, figure 1(a) suggests that there is a random orientation of grains. In comparison, the (111) and (220) peaks of the copper rod material are less pronounced (figure 1(b)), suggesting there could be some texture resulting from the initial forming of the rod stock.

After rolling, x-ray 2θ scans of the copper rod and copper plate were taken at each deformation level. Figure 2(a) shows that for the copper rod samples, with increasing deformation, the (220) orientation dominates, while the non-(220) peaks are greatly reduced. A reduction in secondary peaks occurs at each additional level of deformation. Figure 2(b) shows no

Table 1. Deformation and annealing conditions for the starting rod and plate samples. A back pressure of 2–6 μ Torr and a partial pressure of Ar/H₂ 5% at 200 mTorr were used.

Reduction (%)	95.5	97.5	98.5	99	99.5	Duration (h)
Rod thickness (μ m)	310	170	130	67	36	N/A
Plate thickness (μ m)	95	58	39	20	10	N/A
	As-rolled	As-rolled	As-rolled	As-rolled	As-rolled	N/A
Annealing temperature ($^{\circ}$ C)	300	300	300	300	300	6
	500	500	500	500	500	1
	550	550	550	550	550	1
	600	600	600	600	600	1
	650	650	650	650	650	1
	700	700	700	700	700	1

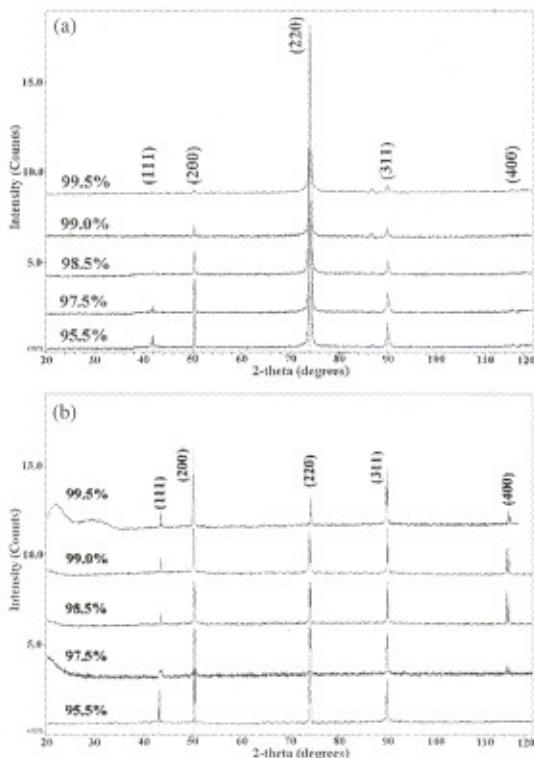


Figure 2. Deformation texture of rolled copper samples.
(a) 2θ scans for the copper rod after rolling at increasing levels of deformation showing increasing intensity of the (220) peak,
(b) 2θ scans of the copper plate after rolling with no indication of a dominant peak.

predominant peak, and no discernable trend with increasing levels of deformation. It appears that the initial texture in the copper plate samples is retained even after significant amounts of deformation.

Following the heat treatments, x-ray 2θ scans were taken. Figure 3 shows that a sharp cube (200) recrystallization texture developed in the copper rod samples annealed at 750 $^{\circ}$ C for 1 h. Note that a sharp cube texture was formed for all levels of deformation from 95.5 to 99.5%. In order to gauge the effect of annealing temperature on in-plane and out-of-plane alignment,

Φ scans and Ψ scans were conducted. Figure 4 shows how the Φ scan full width half maximum (FWHM) values change with temperature for the copper rod samples that had been

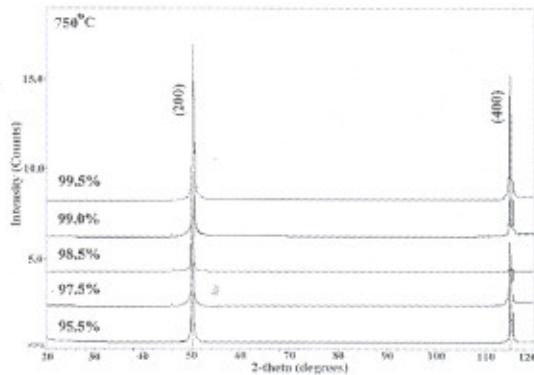


Figure 3. Texture development in the cold rolled copper rod samples annealed at 750 $^{\circ}$ C showing the dominance of the (200) orientation at all deformation levels.

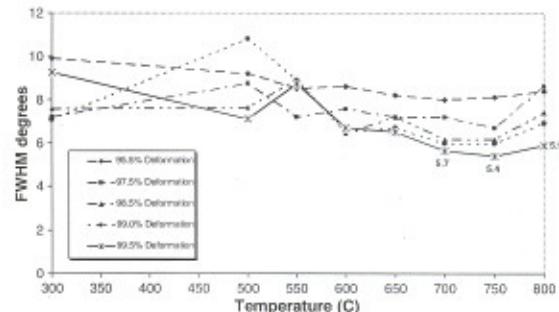


Figure 4. Effect of annealing temperature on the Φ scan full width half maximum (FWHM) values for all of the copper rod samples.

rolled to different amounts of deformation. The sharpest Φ scan peak was obtained at a deformation level of 99.5% and annealing temperature of 750 $^{\circ}$ C, with an FWHM intensity value of 5.4 $^{\circ}$. Figure 5 shows under these deformation and annealing conditions that Φ and Ψ scan FWHM values were 5.4 $^{\circ}$ and 5.8 $^{\circ}$. Microtexture measurements using orientation imaging microscopy (OIM) on this sample revealed that the misorientation between adjacent grains was $<10^{\circ}$ for more than 97.5% of grains in the sample. No significant twinning was observed under a scanning electron microscope (see figure 6).

The surface roughness of the copper tapes was slightly higher than desirable, but determined to be largely a result of the final rolls used. The final rolls had not been polished for an

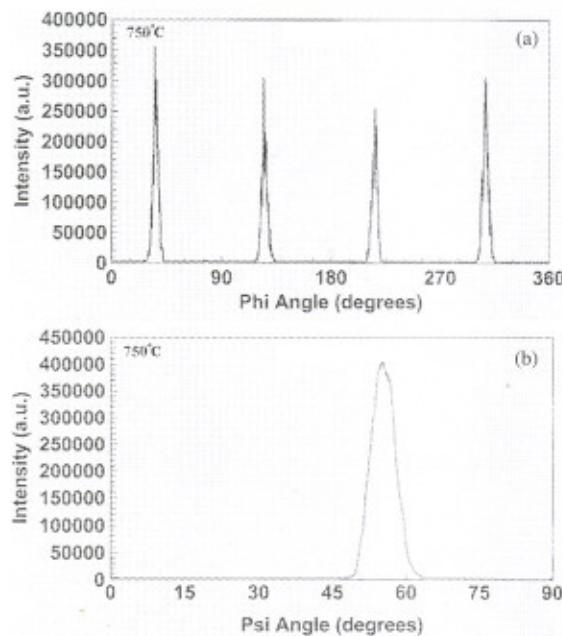


Figure 5. Φ and Ψ scans for the copper rod samples annealed at 750 °C after deformation to 99.5% reduction. (a) Φ scan with FWHM value of 5.4°, and (b) Ψ scan with FWHM value of 5.8°.

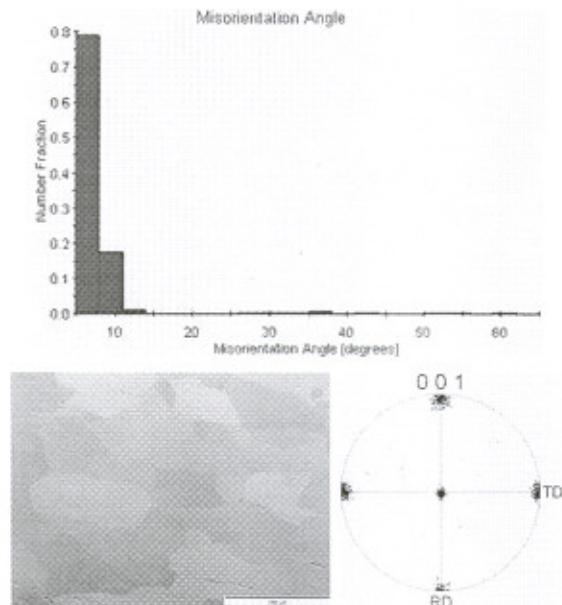


Figure 6. OIM microtexture results with pole figure reveal more than 97.5% of grains with $<10^\circ$ misorientation. SEM micrograph of microstructure showing a stable equiaxed grain structure at 750 °C with no observable twinning.

(This figure is in colour only in the electronic version)

extended period of time, resulting in initial surface roughnesses of the copper substrate to be typically between 20 and 30 nm rms with occasional samples <20 nm rms as determined from AFM measurements. The rolls used in the final deformation

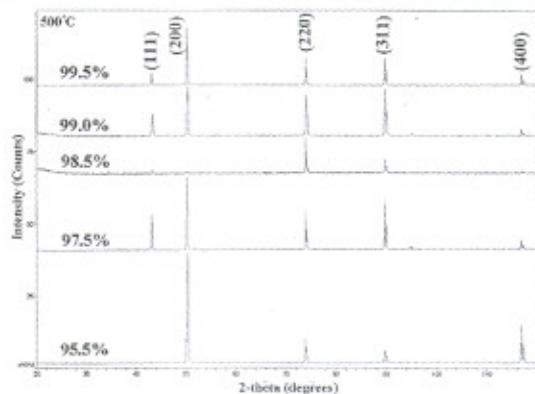


Figure 7. Texture development in the cold rolled copper plate samples annealed at 500 °C showing several FCC peaks.

have been removed for surface polishing and the use of the newly polished rolls on additional copper substrates resulted in surface roughness <10 nm rms for all samples.

X-ray 2θ scans were also taken of the copper plate after heat treatment. A clear (200) recrystallization texture did not develop in any of the deformed and annealed samples. Perhaps the best texture development in the copper plate samples occurred at 500 °C (figure 7). In comparison to figure 3, which shows the 2θ scans for the rod samples, note that though the (200) peak is the most intense, other peaks are also observed. Since no trend or significantly predominant peaks were seen, additional characterization was not performed on the copper plate samples.

4. Summary and conclusions

Cold rolling and annealing experiments carried out on two materials with the same initial composition, but different prior deformation history, resulted in very different texture in the processed samples. Though there appeared to be no significant initial texture in both the extruded rod and rolled plate materials, after heavy levels of deformation the copper rod developed a (220) deformation texture while the copper plate did not. Subsequent annealing of the copper rod resulted in the formation of a (200) recrystallization texture. The best biaxial alignment occurred at 750 °C and 99.5% deformation. On the other hand, the texture of the copper plate samples showed no trends during deformation or preferred orientation after annealing. Since the initial copper rod and plate were obtained as-produced, without annealing, the difference is likely to be due to the initial cold work performed prior to the deformation process conducted above: in particular, the drawing of the rod versus the rolling of the plate.

References

- [1] Diaz J, Segarra M, Espiell F and Piñol S 2001 *Supercond. Sci. Technol.* **14** 576–82
- [2] Goyal A *et al* 1997 *J. Mater. Res.* **12** 2924–40
- [3] Norton D P *et al* 1996 *Science* **274** 755
- [4] He Q, Christen K K, Budai J D, Specht E D, Lee D F, Goyal A, Norton D P, Paranthaman M, List F A and Droeger D M 1997 *Physica C* **275** 155

- [5] Paranthaman M *et al* 1997 *Physica C* **275** 266
- [6] Goyal A *et al* 2002 *Physica C* **382** 251–62
- [7] Lehnstorff B, Hörtig M, Monter B, Piel H, Pouryamout J, Pupeter N and Bischoff E 1999 *Proc. 4th European Conf. on Applied Superconductivity (Spain)*
- [8] Nekkanti R M *et al* 2001 *IEEE Trans. Appl. Supercond.* **11** (1)
- [9] de Boer B, Reger N, Fernandez G R, Eickemeyer J, Berberich P, Prusseit W, Holzapfel B and Schultz L 2001 *IEEE Trans. Appl. Supercond.* 3477–80
- [10] Jin M, Han S-C, Sung T-H and No K 2000 *Physica C* **301** 104
- [11] Tuissi A, Corti R, Villa E, Bramley A P, Vickers M E and Evetts J E 1999 *EUCLAS '99: Proc. 4th European Conf. on Applied Superconductivity (Barcelona, 1999) (Inst. Phys. Conf. Ser. 167)* (Bristol: Institute of Physics Publishing) p 399
- [12] Bhattacharya R N, Chen J, Spagnol P and Chaudhuri T 2004 Electrodeposited biaxially textured Ni layer for YBCO superconductor oxide films *J. Electrochem. Soc.* submitted